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## 塗膜電磁鋼片退火附著性之研究 Investigations on the Post-Annealing Adhesion Capability of Coated Electrical Steels

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### Abstract

Electrical steels (ES) are the core for electrical motors, power generators, etc. To restore the electromagnetic property of ES after stamping, stress-relieving annealing is required.

However, annealing can be very destructive to the insulation coating on ES and an inappropriate annealing usually results in coating adhesion failure, which badly affects the performance of ES facilities.

Therefore, effects of the atmosphere in a practical (not absolutely air-tight) furnace on the post-annealing adhesion capability of ES coating were investigated to qualitatively reveal the influences of O<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>, and H<sub>2</sub>O, respectively. The presence of CO<sub>2</sub> has little effect while both O<sub>2</sub> and H<sub>2</sub>O can deteriorate the post-annealing adhesion capability. Contrarily, H<sub>2</sub> has protecting effects so that good post-annealing adhesion capability of coating can be fulfilled. Therefore, to ensure good post-annealing adhesion capability to the coating, minimal contents of oxygen and humidity in the furnace atmosphere is required and the presence of some hydrogen in the furnace atmosphere can be helpful.

**Keywords:** Electrical steel; Annealing; Coating; Adhesion; Atmosphere.

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## 1. Introduction

Electrical steels (ES), or silicon steels, are an important family of steels that consist of Fe-Si alloys. Such alloys have special electromagnetic properties and are the core of motors, power generators, compressors, and transformers. And the properties of electrical steel are very critical to the efficiency of energy transformation.

The loss during the energy transformation (i.e., the “iron loss”) is composed of “the hysteresis loss” and “the Eddy current loss”<sup>[1]</sup>. The former is an intrinsic property and is mainly controlled by the alloying composition and process history of electrical steels. On the other hand, the latter depends more on extrinsic parameters, e.g., the design of the energy-transforming systems and the geometry of ES core. Eddy current loss is proportional to the square of thickness, therefore electrical steels are arranged as a stack of thin sheets. Besides, Eddy current loss is also inversely proportional to the interlayer resistivity between ES sheets. Therefore most modern ES coils are coated with an insulating layer<sup>[2,3]</sup>.

Since ES are used in stacks of predesigned stamped shapes, ES coils must be slit in to hoops of the required width first. Then the hoop runs through a high-speed punching process to form shaped stampings. However, the punching process leaves residual stress on the thus formed stampings. Therefore, electromagnetic properties (e.g., the iron loss) of ES always deteriorate after the punching process. To eliminate the residual stress and to restore the electromagnetic properties of the ES stampings, thermal treatment (typically 700~850°C) is required after the punching process. And such kinds of thermal treatment are termed as “stress-relieving annealing

(SRA)”<sup>[4,5]</sup>.

Practically, lubricant oil must be applied in the punching process of ES. Therefore, the punched ES stampings are covered by oil. Such residual oil can be problematic and needs to be removed before SRA. For that, a “burn-off” process by heating ES stampings in air is usually carried out before SRA. As compared with the annealing process, the required burn-off temperature is significantly lower (typically 250°C~500°C).

Though thermal treatments as described above can recover and even improve the electromagnetic properties of ES stampings after the punching process, it can also be very destructive to the insulating coating on ES. Inappropriate thermal treatments often lead to failures in coating adhesion and interlayer resistance (electrical insulation between adjacent stampings). Ultimately, such failures would result in serious problems such as increased iron loss, lowered efficiency, and abnormal temperature rise for ES facilities. In other aspects, parameters (e.g., the furnace atmosphere, the heating profile, etc.) and equipment for thermal treatment are so diversified that the result of annealing can vary considerably, especially in the coating adhesion capability and the appearance of annealed stampings. Hence, the SRA process is the most difficult to control among all the working processes of ES. And in many cases, failures were resulted from variations or deviations of the above thermal treatment parameters<sup>[6,7]</sup>.

Therefore, effects of furnace atmosphere (O<sub>2</sub>, H<sub>2</sub>O, H<sub>2</sub>, and CO<sub>2</sub>) on the post-annealing adhesion capability of ES coating were investigated in this study. In addition, influences of ES substrate (in view of the major alloying compositions for different

grades of ES) were also studied. And results from this study can be guidelines to optimize the SRA process of ES.

## 2. Experiments

### 2.1 The materials

Three grades of electrical steel were used as the substrate. The major alloying elements (Si and Al) for these substrates are shown in Table 1. The Si and Al contents are ca 3.1~1.9% and ca 1.1~0.3%, respectively. For the coated samples, substrates were coated with a waterborne chromium-free paint and then baked by heating, after which the coating can be cured. And the thus obtained ES samples are coded as S-1, S-2 and S-3, respectively. °

Table 1 Composition of the major alloying elements in the ES substrates of the three samples.

		Samples		
		S-1	S-2	S-3
Substrate	Si	ca 3.1%	ca 3.0%	ca 1.9%
	Al	ca 1.1%	ca 0.6%	ca 0.3%

### 2.2 Anti-corrosion capability

The anti-corrosion capability of the investigated coated ES (before thermal treatment) was evaluated on edge-sealed samples, following the ASTM-B117 method (the neutral salt spray test, SST). The salt spray was produced from 35°C 5% NaCl aqueous solution at a rate of 1~2 mL/hr. The anti-corrosion capability of samples was then judged by the rusting area percentage after 16hrs SST.

### 2.3 Thermal treatment (Burn-off & SRA)

Thermal treatments were performed in a single-chamber furnace. This furnace is “NOT airtight”, therefore nitrogen of high flow rate (>130L/min) is required to apparently repel air out of the chamber, but trace of oxygen may still remain inside nevertheless. If the nitrogen flow rate is not high enough, air (contains oxygen and humidity) can significantly leak into the furnace so that surface oxidation can be very recognizable for annealed samples.

A 7-stage heating profile was adopted for the thermal treatment (Figure 1). Stages 1 and 2 consist of the “burn-off period” and stages 3~7 function as the “stress-relieving annealing (SRA) period”. The maximal temperature for burn-off and annealing are 275°C and 780°C, respectively. Table 2 shows the furnace atmosphere for the above thermal treatment process. Six different combinations of atmospheres (I ~ VI) were tested. Burn-off was carried out “in ambient air” for Atmospheres II ~ VI. As a comparison, burn-off stage for Atmosphere I was done in nitrogen atmosphere. And for SRA stages, five kinds of atmosphere were tested: N<sub>2</sub> (neutral), O<sub>2</sub>-containing N<sub>2</sub> (oxidative), H<sub>2</sub>O-containing N<sub>2</sub> (wet), H<sub>2</sub>-containing N<sub>2</sub> (reductive), and CO<sub>2</sub>-containing N<sub>2</sub>. In the furnace, two configurations for positioning the samples were adopted. Samples were “individually isolated” or “stacked by a pressurized fixture” to simulate the most outer and the inner stampings of ES cores. And for each test, isolated and stacked samples were annealed simultaneously in the same batch of thermal treatment.

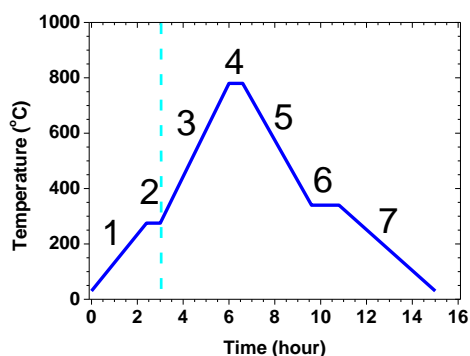


Figure 1 The heating profile for the investigated thermal treatments.

## 2.4 Adhesion capability of coating for annealed samples

After thermal treatment, adhesion capability of

the coating to substrate was evaluated by Scotch tape (type 600). Upon fast removal of the tape from the annealed sample, clean tape without stain and intact surface without substrate exposure stands for excellent adhesion of the coating to the substrate. On the contrary, if coating adhesion deteriorates significantly after thermal treatment, exposure of bare substrate (peeling-off of the coating) and stains on tape would take place. And the adhesion capability of coating after thermal treatment can be differentiated by the extent of substrate exposure and tape stains.

Table2 Furnace atmospheres for the thermal treatment in Figure 1.

Furnace atmosphere code #	Stage of the thermal treatment and the used atmosphere						
	Burn-off		Stress-relieving annealing (SRA)				
	1	2	3	4	5	6	7
I	Neutral N <sub>2</sub>		Neutral N <sub>2</sub> (145L/min)				
II	Air		Neutral N <sub>2</sub> (145L/min)				
III	Air		Oxidative N <sub>2</sub> (105L/min, to allow air leakage )				
IV	Air		Wet N <sub>2</sub> (135L/min, flow passing over 20°C water surface)				
V	Air		Reductive N <sub>2</sub> (130L/min N <sub>2</sub> + 20L/min H <sub>2</sub> )				
VI	Air		CO <sub>2</sub> -containing N <sub>2</sub> (125L/min N <sub>2</sub> + 25L/min CO <sub>2</sub> )				

## 2.5 Cross-sectional analysis of the coatings

Specimens were first vapor deposited by a thin layer of platinum (Pt). Then the surface was milled by dual-beam focused gallium ion beams (DB-FIB) to cut out a rectangular hole, from which cross sectional images can then be taken by scanning electron microscopy (SEM) at a magnification of 65,000X. And the distribution of elements over cross

sections can be revealed by mapping (the energy dispersion spectroscopy, EDS): Fe map for the substrate, P map for the coating, and O map for evaluating the extent of oxidation.

## 3. DISCUSSION

In the thermal treatment processes, ES stampings either expose directly to the furnace atmosphere (the most outer surface) or contact

adjacent sheets (probably with pressure, the inner parts). The results of annealing can be different for the outer and the inner parts of an annealed ES core. Therefore, two configurations for positioning samples in the furnace were studied to simulate the most outer and the inner stampings of annealed ES cores.

### 3.1 Samples annealed in the “individually isolated” configuration







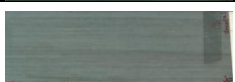
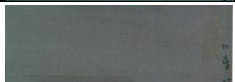

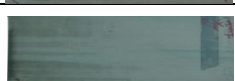
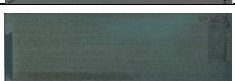
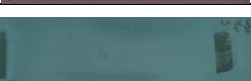






Annealing in this configuration was adopted to investigate effects of furnace atmosphere on “the most outer stampings” of ES cores in thermal treatments.

After thermal treatment (Figure 1 and Table 2), Scotch 600 tape was attached with finger pressure onto each sample and then torn off rapidly to evaluate the adhesion capability of the coating. Table 3 shows the scan images of the ES samples annealed in the “individually isolated” configuration after the tape

test.

The most obvious result in Table 3 is that the appearance of annealed samples can differ so much with the furnace atmosphere. In addition, for a specific SRA atmosphere, the post-annealing appearance of samples also depends on the substrate, suggesting that the alloying composition of ES should have effects. For a specific substrate, samples annealed in Atmospheres I and II (burn-off: air or N<sub>2</sub>; SRA: both N<sub>2</sub>) bear close resemblance. Obviously, burn-off at 275°C in neutral N<sub>2</sub> and in air should have similar effect. Interestingly, dark and uniform appearance is provided by the thermal treatment in Atmosphere V (H<sub>2</sub>-containing nitrogen, reductive) for all the samples. As compared with other Atmospheres, the distinctness of Atmosphere V reveals that atmosphere-induced oxidation plays a key role in the thermal treatment process of ES samples.




Table3 Appearance of the ES samples annealed in the “individually isolated” configuration after the tape test (Scanned images: black belts in the images are caused by the scanner but not due to the samples).

Furnace atmosphere			Annealed samples after the tape test		
			S-1	S-2	S-3
I	N <sub>2</sub>	N <sub>2</sub>			
II	Air	N <sub>2</sub>			
III	Air	N <sub>2</sub> + O <sub>2</sub>			
IV	Air	N <sub>2</sub> + H <sub>2</sub> O			
V	Air	N <sub>2</sub> + H <sub>2</sub>			
VI	Air	N <sub>2</sub> + CO <sub>2</sub>			

All samples annealed in Atmosphere III ( $O_2$ -containing nitrogen, oxidative) revealed very different appearance from Atmospheres I and II. Annealed samples S-1 and S-2 are of pale color, whilst sample S-3 is reddish. Undoubtedly, such results should be related to the different chemical reactivity (i.e., resistance to oxidation) of the three grades of ES substrate. Since the chemical inertness of ES substrates does affect the anti-corrosion capability of coated ES, the salt spray test (SST, 16hrs)

was also done on the three samples (as received, before thermal treatment). As shown in Table 4, both samples S-1 and S-2 show only few rust spots after 16hrs SST, whilst significant corrosion has occurred in Sample S-3 after the same test. Amazingly, Tables 3 and 4 reflect a clear correspondence between results of SST and oxidative SRA (Atmosphere III). This suggests SST can probably be an alternative to evaluate the capability of coated ES for SRA in oxidative atmospheres.

Table4 Salt spray test (SST, 16hrs) results for the investigated coated ES samples (Scanned images).

S-1	S-2	S-3
		

SRA in the atmosphere IV (wet nitrogen, dew point: ca +20°C) turned all the samples into grey appearance.

After SRA in the Atmosphere VI ( $CO_2$ -containing nitrogen atmosphere), Samples S-1 and S-2 also show pale color, whilst S-3 is somewhat lavender. Interestingly, samples annealed in Atmosphere VI and III bear some resemblance.



















Table 5 compares the tapes that were torn from annealed samples in the tape test (Tables 3 and 5 are not on the same scale). Most of the torn tapes are

clean without any de-attachment or contaminations. However, tapes for Atmosphere V ( $H_2$ -containing nitrogen, reductive) have carbon-like stains. Since there is no substrate exposure after tape test, stains on tape should be from the very surface of the annealed coating, while the main body of coating still adheres firmly to the substrate. This result can be attributed to the coking process of coating, in which oxidative pyrolysis loss of organic components is suppressed due to the presence of  $H_2$ . For Atmospheres I and II (neutral SRA atmosphere), all the annealed samples

show neither substrate exposure (no peeling off for the coating after tape tearing) nor stains on the torn

tapes, revealing good post-annealing adhesion capability in these two conditions.

Table5 Tapes torn away from the ES samples annealed in the “individually isolated” configuration (scanned images).

Furnace atmosphere			Tapes torn from the annealed samples		
			S-1	S-1	S-1
I	N <sub>2</sub>	N <sub>2</sub>			
II	Air	N <sub>2</sub>			
III	Air	N <sub>2</sub> + O <sub>2</sub>			
IV	Air	N <sub>2</sub> + H <sub>2</sub> O			
V	Air	N <sub>2</sub> + H <sub>2</sub>			
VI	Air	N <sub>2</sub> + CO <sub>2</sub>			

For Atmosphere III (O<sub>2</sub>-containing nitrogen, oxidative), coatings of the annealed Samples S-1 and S-2 still have very good post-annealing adhesion capability. However, Sample S-3 annealed in Atmosphere III shows moderate substrate exposure (coating peeled) in the tape test. An even worse result for Sample S-3 is obtained in Atmosphere IV (wet nitrogen, dew point: ca +20°C): the coating is torn away completely in the tape test and the substrate thus exposed is deep dark without gloss (probably due to the formation of Fe<sub>3</sub>O<sub>4</sub> that resulted from the reaction between iron and humidity at high temperatures). On the contrary, Samples S-1 and S-2 annealed in Atmosphere IV still show good results. And this clearly reveals that substrate is also an

affecting factor to the annealing capability for the coated ES products.

Samples S-1, S-2 and S-3 annealed in Atmosphere VI (CO<sub>2</sub>-containing nitrogen) all retain good post-annealing adhesion capability for the coating. It follows that the influence of CO<sub>2</sub> on these samples should be insignificant or in a minor extent. As compared with Atmosphere II (neutral nitrogen) and III (O<sub>2</sub>-containing nitrogen), Sample S-3 annealed in Atmosphere VI showed color in between. Therefore, Atmosphere VI should induce a degree of oxidation slightly higher than Atmosphere II but much less than Atmosphere III. The above tape test results for ES samples annealed in the “individually isolated” configuration are summarized in Table 6.

Table6 Summary of the tape test results for samples annealed in the “individually isolated” configuration.

	Furnace atmosphere			Tape test results		
				S-1	S-2	S-3
Substrate appearance	I	N <sub>2</sub>	N <sub>2</sub>	◎	◎	◎
	II	Air	N <sub>2</sub>	◎	◎	◎
	III	Air	N <sub>2</sub> + O <sub>2</sub>	◎	◎	X
	IV	Air	N <sub>2</sub> + H <sub>2</sub> O	◎	◎	XX
	V	Air	N <sub>2</sub> + H <sub>2</sub>	◎	◎	◎
	VI	Air	N <sub>2</sub> + CO <sub>2</sub>	◎	◎	◎
Tape appearance	I	N <sub>2</sub>	N <sub>2</sub>	◎	◎	◎
	II	Air	N <sub>2</sub>	◎	◎	◎
	III	Air	N <sub>2</sub> + O <sub>2</sub>	◎	◎	X
	IV	Air	N <sub>2</sub> + H <sub>2</sub> O	◎	◎	XX
	V	Air	N <sub>2</sub> + H <sub>2</sub>	X	XX	XX
	VI	Air	N <sub>2</sub> + CO <sub>2</sub>	◎	◎	◎
Substrate : ◎intact → ○slightly peeled → △locally peeled → X extendedly peeled → XX completely peeled Tape : ◎no stain → ○slightly stained → △locally stained → X extendedly stained → XX completely stained						

### 3.2 Samples annealed in the “stacked by a pressurized fixture” configuration

Annealing in this configuration was adopted to investigate effects of furnace atmosphere on “the inner stampings” of ES cores in thermal treatments.

Table 7 shows the ES samples annealed in this configuration after the tape test. Tapes torn from these annealed ES samples are compared in Table 8 (Tables 7 and 8 are not on the same scale). And the tape test results are summarized in Table 9.

As compared with the “individually isolated” configuration (Tables 3, 5 and 6), samples annealed as a pressured stack show surprisingly little difference in the appearance and in the adhesion capability of the coating. All the annealed samples in this configuration are deep dark, regardless of

furnace atmosphere and substrate grade. And the post-annealing adhesion capability of the coating is also good. Please recall that Sample S-3 annealed as “individually isolated” specimens in Atmospheres III (O<sub>2</sub>-containing nitrogen, oxidative) and IV (wet nitrogen, dew point: ca +20°C) shows poor tape test results (Tables 3, 5, and 6). Contrarily, Sample S-3 annealed as “a pressured stack” in these two atmospheres has good post-annealing adhesion capability for the coating (Tables 7~9). In addition, there are more or less carbon-like stains on the tapes torn from samples annealed in this configuration. Such results suggest that the oxidative pyrolysis loss of organic components in the coating had been somewhat suppressed, probably due to the unavailability of oxidants (i.e., O<sub>2</sub> and H<sub>2</sub>O) within the pressured stack.



The above results are consistent with practical experiences. The inner part of annealed ES cores always has better batch-to-batch consistency than the outer stampings. Undoubtedly, stampings in well-pressured stack are protected from the furnace atmosphere so that the atmosphere-induced reactions (e.g. oxidation) in the coating and on the ES substrate surface can be inhibited. Therefore, for ES stampings within a well-stacked core, thermal treatments should

only lead to coking of the coating itself and the chemistry of ES substrate surface should not change much. However, for the outer part of ES core, chemical reactions with atmosphere are more liable to occur. Therefore, for the outer part of ES core, careful control over thermal treatment parameters (atmosphere, temperature, time...etc.) is required for better batch-to-batch consistency in the appearance and the coating adhesion capability.

Table7 Appearance of the ES samples annealed in the “stacked by a pressurized fixture” configuration after the tape test (Scanned images: black belts in the images are caused by the scanner but not due to the samples).



















Furnace atmosphere			Annealed samples after the tape test		
			S-1	S-2	S-3
I	N <sub>2</sub>	N <sub>2</sub>			
II	Air	N <sub>2</sub>			
III	Air	N <sub>2</sub> + O <sub>2</sub>			
IV	Air	N <sub>2</sub> + H <sub>2</sub> O			
V	Air	N <sub>2</sub> + H <sub>2</sub>			
VI	Air	N <sub>2</sub> + CO <sub>2</sub>			

Table8 Tapes torn away from the ES samples annealed in the “stacked by a pressurized fixture” configuration (scanned images).



















Furnace atmosphere			Tapes torn from the annealed samples		
			S-1	S-2	S-3
I	N <sub>2</sub>	N <sub>2</sub>			
II	Air	N <sub>2</sub>			
III	Air	N <sub>2</sub> + O <sub>2</sub>			
IV	Air	N <sub>2</sub> + H <sub>2</sub> O			
V	Air	N <sub>2</sub> + H <sub>2</sub>			
VI	Air	N <sub>2</sub> + CO <sub>2</sub>			

Table9 Summary of the tape test results for samples annealed in the “stacked by a pressurized fixture” configuration.

	Furnace atmosphere			Tape test results		
				S-1	S-2	S-3
Substrate appearance	I	N <sub>2</sub>	N <sub>2</sub>	◎	◎	◎
	II	Air	N <sub>2</sub>	◎	◎	◎
	III	Air	N <sub>2</sub> + O <sub>2</sub>	◎	◎	◎
	IV	Air	N <sub>2</sub> + H <sub>2</sub> O	◎	◎	◎
	V	Air	N <sub>2</sub> + H <sub>2</sub>	◎	◎	◎
	VI	Air	N <sub>2</sub> + CO <sub>2</sub>	◎	◎	◎
Tape appearance	I	N <sub>2</sub>	N <sub>2</sub>	◎	◎	○
	II	Air	N <sub>2</sub>	○	◎	△
	III	Air	N <sub>2</sub> + O <sub>2</sub>	◎	◎	○
	IV	Air	N <sub>2</sub> + H <sub>2</sub> O	○	◎	△
	V	Air	N <sub>2</sub> + H <sub>2</sub>	X	XX	XX
	VI	Air	N <sub>2</sub> + CO <sub>2</sub>	◎	◎	◎

Substrate :  
 ◎intact → ○slightly peeled → △locally peeled → X extendedly peeled → XX completely peeled

Tape :  
 ◎no stain → ○slightly stained → △locally stained → X extendedly stained → XX completely stained

### 3.3 Effects of furnace atmosphere on the microstructure of coating

In previous sections, the adhesion capability of coating for samples annealed in different atmospheres has been revealed. Table 6 clearly shows that furnace atmosphere does have effects, but to what extent it can influence indeed depends on the ES substrate (the alloying composition). Among the three samples, S-3 is the most sensitive to the variation of furnace atmosphere. On the contrary, Samples S-1 and S-2 always have good post-annealing adhesion capability, regardless of the furnace atmosphere adopted in the thermal treatments.

To further clarify, FIB/SEM/EDS analyses were carried out on samples annealed in the “individually isolated” configuration. And key results are shown in Table 10 and 11 for Atmospheres II (neutral & dry nitrogen) and IV (wet nitrogen, dew point: ca +20°C), respectively. For all the images, it is the middle layer that presents the cross section of the coating. The top dense layer is the protective Pt film deposited prior to DB-FIB milling. And the bottom dense layer is the ES substrate.

All samples annealed in Atmosphere II show well-defined cross sections, in which the interface between coating and substrate is clear-cut. This suggests that the chemistry of substrate surface was changed little by the thermal treatment in Atmosphere II. Samples S-1 and S-2 annealed in Atmosphere IV also have clear cross sections. However, the microstructure of Sample S-3 annealed in Atmosphere IV is much more complicated and blurry. Comparing P maps of Sample S-3 annealed in Atmospheres II and IV, the coating layer is obviously

thickened by the annealing in Atmosphere IV. Besides, Fe, P, and O maps of Sample S-3 annealed in Atmosphere IV clearly reveal the formation of an “oxidation zone on the substrate surface” (beneath the coating, i.e., the inner oxidation zone, IOZ) after this thermal treatment. These results could imply that the substrate of Sample S-3 kept reacting with the coating as well as the atmosphere for the duration of the thermal treatment in Atmosphere IV.

From Tables 3, 5 and 6, Sample S-3 annealed in Atmosphere IV completely failed the tape test whereas that annealed in Atmosphere II still retained good adhesion capability. Table 10 shows that Sample S-3 annealed in Atmosphere II have no IOZ structure. Moreover, Samples S-1 and S-2 annealed in Atmosphere IV, which still have good coating adhesion capability after the thermal treatment, don't have the IOZ structure either (Table 11). Undoubtedly, the adhesion failure of Sample S-3 annealed in Atmosphere IV should be attributed to the humidity-induced formation of IOZ in the surface of ES substrate (beneath the coating).

The formation of IOZ in ES has been reported for bare substrates by other groups [8]. And we have also found that failure in the post-annealing adhesion capability of coating can result from the formation of extended IOZ in coated ES that was annealed inappropriately [6,7]. In such cases, adhesion failure occurs on the “interface between the IOZ of substrate and the underlying un-oxidized ES” (interface located “within the substrate”), instead of the “interface between the coating and the substrate”. And it is “the substrate IOZ attached to the coating” that is torn from the annealed specimen in the tape

test.

The above findings reveal that both the furnace atmosphere and the ES alloying composition can be critical to the formation of IOZ in ES, which in turn will significantly affect the post-annealing adhesion capability of coating. And the operation window in furnace atmosphere is dependent on the ES substrate of stampings that are going to be annealed. Therefore, optimization of furnace atmosphere by trial runs should be required for ES of different substrate

grades to guarantee the quality of thermal treatment results.

This preliminary study has qualitatively provided information on how the atmosphere in furnaces that are not air-tight influences the post-annealing adhesion capability of coating to ES substrate. Quantitative influences of the atmosphere in ideal (absolutely air-tight) furnaces and their synergistic effects on thermal treatments is under investigation.

Table10 FIB, SEM, and EDS mapping of samples annealed in the “individually isolated” configuration with Atmosphere II.

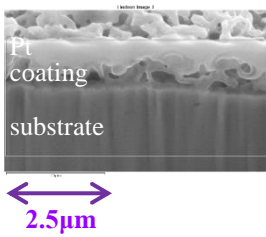


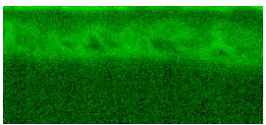
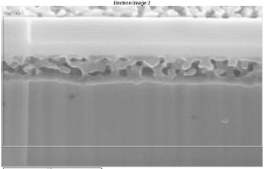


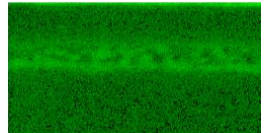
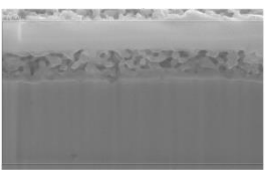
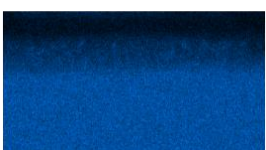

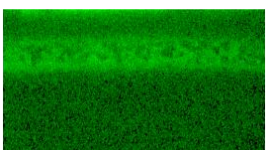
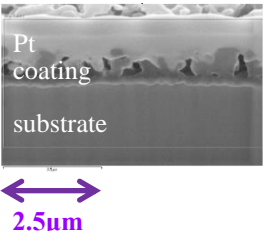
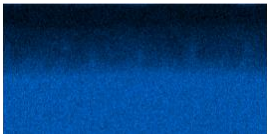
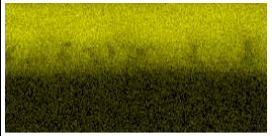
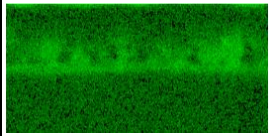
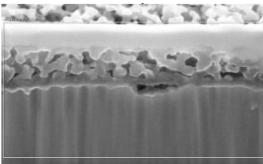
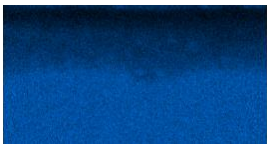

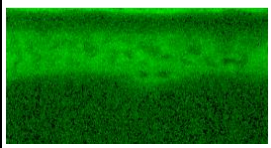
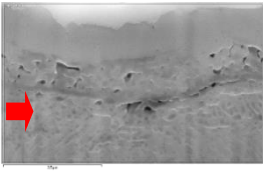

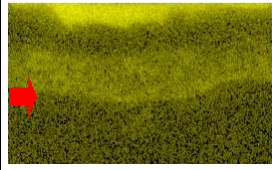
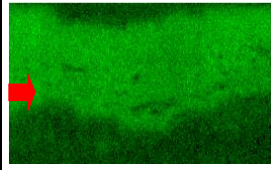
	FIB / SEM 65,000X	EDS mapping		
		Fe	P	O
S-1				
S-2				
S-3				

Table11 FIB, SEM, and EDS mapping of samples annealed in the “individually isolated” configuration with Atmosphere IV Arrows indicate the presence of inner oxidation zone formed in Sample S-3 after the thermal treatment.

	FIB / SEM 65,000X	EDS mapping		
		Fe	P	O
S-1				
S-2				
S-3				

#### 4. CONCLUSIONS

Our investigations have qualitatively revealed how the atmosphere in furnaces (for those that are not absolutely air-tight) affects the post-annealing adhesion capability of the insulation coating to ES substrate. The outer stampings of ES stacks are much more liable to be affected by the atmosphere than the inner part. The presence of CO<sub>2</sub> has little effect and may be regarded as a neutral component in the atmosphere. Both O<sub>2</sub> and H<sub>2</sub>O are oxidative and can deteriorate the post-annealing adhesion capability. But to what extent they can influence is whereas substrate-dependent. Upon inappropriate thermal treatments, the substrate surface (beneath the coating) can be oxidized extendedly, due to which the post-annealing adhesion capability for the coating is fatally affected. Contrarily, H<sub>2</sub> has protecting effects

so that good post-annealing adhesion capability of coating can be fulfilled regardless of the ES substrate grades. Therefore, to ensure good post-annealing adhesion capability to the coating, contents of oxygen & humidity (dew point) in the furnace atmosphere should be minimized or at least kept at low levels. In addition, it is very helpful to introduce some hydrogen into the furnace atmosphere.

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